IS 11255-4 (2006): Methods for measurement of emissions from stationary sources, Part 4: Hydrogen sulphide and carbon disulphide [CHD 32: Environmental Protection and Waste Management]

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भारतीय मानक

स्थिर स्रोतों से उत्सर्जन को मापने की पद्धति

भाग 4 हाईड्रोजन सल्फाईड और कार्बन डाइसल्फाईड

(पहला पुनरीक्षण)

Indian Standard

METHOD FOR MEASUREMENT OF EMISSION FROM STATIONARY SOURCES

PART 4 HYDROGEN SULPHIDE AND CARBON DISULPHIDE

(First Revision)

ICS 13.040.40

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 3

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environment Protection and Waste Management Sectional Committee had been approved by the Chemical Division Council.

Carbon disulphide and hydrogen sulphide are emitted together in processes like cellulosic fibre manufacture. This standard prescribes a method for determination of these gases in presence of one another.

This standard was originally published in 1985. With a view to upgrade the standard based on the technological developments in this area, this revision is brought out incorporating the details of the apparatus required for testing and calculations for more clarity and better accuracy. The precision of the methods are yet to be established. Aqueous tension at various temperatures are also incorporated in the standard.

There is no ISO Standard on the subject. The standard has been developed based on the indigenous methods available in India.

The composition of the Committee responsible for the formulation of this standard is given at Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHOD FOR MEASUREMENT OF EMISSION FROM STATIONARY SOURCES

PART 4 HYDROGEN SULPHIDE AND CARBON DISULPHIDE

(First Revision)

1 SCOPE

This standard prescribes a method for the measurement of carbon disulphide and hydrogen sulphide in presence of each other.

2 PRINCIPLE

2.1 Hydrogen sulphide is absorbed in alkaline cadmium chloride solution and the cadmium sulphide formed is estimated iodometrically. The reactions involved are:

$$CdCl_2 + H_2S \longrightarrow CdS + 2HCl$$
$$CdS + 2HCl \longrightarrow CdCl_2 + H2S$$
$$H_2S + I_2 \longrightarrow 2HI + S$$

2.2 Carbon disulphide is absorbed in alcoholic potassium hydroxide solution forming potassium ethylxanthate. The xanthate is estimated iodometrically. The reactions involved are:

$$CS_2 + KOH + C_2H_5OH \longrightarrow KSCSOC_2H_5 + H_2O$$
$$2\,KSCSOC_2H_5 + I_2 \longrightarrow 2KI + (SCSOC_2H_5)_2$$

3 OUTLINE OF METHOD

The air is passed through an absorber train of five absorbers, the first three absorber containing 30 ml each of alkaline cadmium chloride solution and the last two containing 30 ml each of 5 percent alcoholic potassium hydroxide solution. The first three absorbers arrest H_2S and the last two absorbers arrest CS_2 in the sample. After passing the air sample (about 8 litres), the H_2S in the first three absorbers and the CS_2 in the last two absorbers are estimated by treating with standard iodine solution and titrating back the excess iodine with standard sodium thiosulphate solution.

4 APPARATUS

4.1 Absorbers — Five Drechsel bottles (*see* Fig. 1), capacity 250 ml each.

NOTE — Gas scrubbers containing sintered glass plates are not recommended for this method owing to the difficulty of removing the precipitate from the sintered plate.

4.2 Gas Meter — wet type, graduated so that one revolution is equivalent to 0.5 litre.

4.3 Gas Sample Container — of glass, fitted with a single way stopcock on the entry and exit leads.

4.4 Glassware — Erlenmeyer Flask — capacity 500 ml, burette 50 ml, measuring cylinders.

4.5 Wash Bottle

5 REAGENTS

5.1 Cadmium Chloride Solution — Dissolve 10 g of cadmium chloride in 450 ml of distilled water to which 10 ml 0.5 N sodium hydroxide solution is added.

NOTE — Cadmium sulphate solution (5 percent) may also be used, if cadmium chloride is not available.

5.2 Iodine Solution — 0.005 N, standardized against the standard sodium thiosulphate solution.

5.3 Sodium Thiosulphate Solution — 0.005 N, standardized against standard potassium dichromate solution.

5.4 Potassium Dichromate Solution — 0.005 N.

5.5 Potassium Iodide Solution — 20 percent.

Dissolve 20 g of potassium iodide in 100 ml of distilled water.

NOTE — This solution should be prepared fresh.

5.6 Hydrochloric Acid — Concentrated.

5.7 Alcoholic Potassium Hydroxide Solution — Dissolve 5 g of potassium hydroxide in 2-3 ml of water and dilute to 100 ml with absolute ethyl alcohol.

NOTE — This solution should be prepared fresh just before scrubbing the air.

5.8 Acetic Acid — 5 percent.

5.9 Starch Solution — Dissolve about 0.1 g starch in 25 ml hot distilled water and cool before use.

NOTE — This solution is to be prepared fresh.

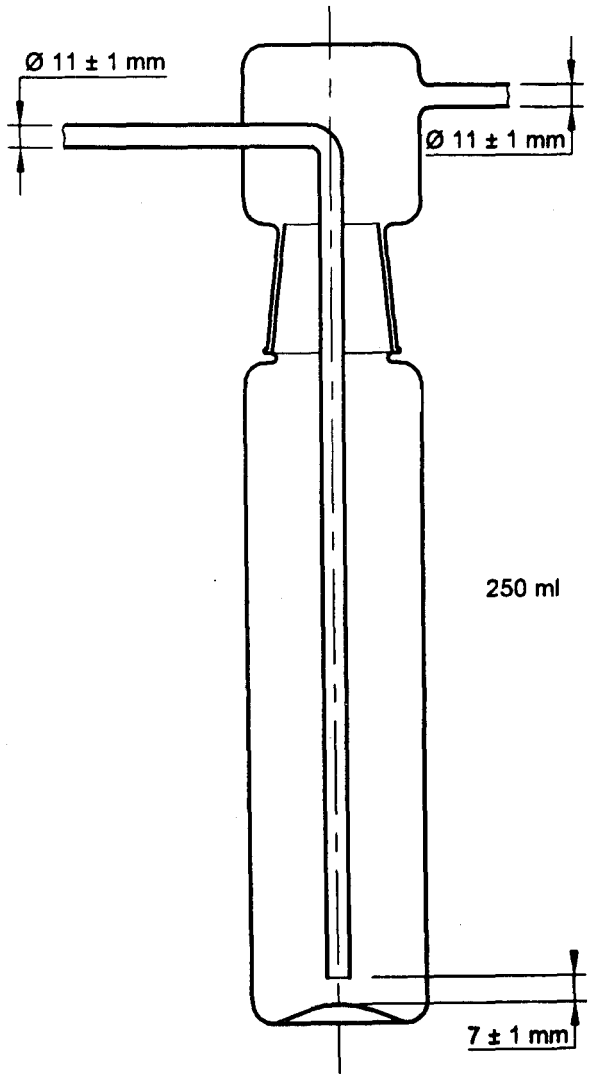

FIG. 1 DRECHSEL BOTTLE

5.10 Phenolphthalein Indicator, 1 percent — Dissolve 1.0 g phenolphthalein in 100 ml of 50 percent ethyl alcohol.

5.11 Sodium Bicarbonate

6 PROCEDURE

6.1 Sampling

Set up the absorber train with five absorbers in series, the first three containing 30 ml of alkaline cadmium chloride solution each and the last two absorbers containing 30 ml of alcoholic potassium hydroxide solution each, with the gas meter to measure the air flow on line. Regulate the flow of the sample at 0.5 litre per minute and pass about 8 litre of sample. Disconnect the sample container and the gas meter. Take the first three absorbers in the train for the estimation of hydrogen sulphide and the last two for the estimation of carbon disulphide.

NOTE — Care has to be taken for exposure to light.

6.2 Standardization of Sodium Thiosulphate Solution

Fill the burette with sodium thiosulphate solution and adjust the liquid level to the zero mark. Put 5-7 ml of 20 percent potassium iodide solution and 10-15 ml of 2 N sulphuric acid using measuring cylinder into 500 ml conical flask. Add 25 ml of potassium dichromate (0.005 N) solution by a transfer pipette, add 0.5 g sodium bicarbonate quickly, cover the flask with a watch glass and leave the mixture for 5 min in a dark place. Then remove the watch glass and rinse it with distilled water into the flask. Dilute the content with distilled water titrate with sodium thiosulphate solution using starch indicator. Calculate the strength of the thiosulphate using the formula:

$$N_1V_1 = N_2V_2$$

where

N_1 = normality of sodium thiosulphate;

V_1 = volume of the thiosulphate consumed for 25 ml of potassium dichromate solution, in ml;

N_2 = normality of potassium dichromate solution; and

V_2 = volume of potassium dichromate solution.

6.3 Determination of the Strength of Iodine Solution

Titrate 25 ml of Iodine solution in a conical flask with standard sodium thiosulphate (0.05 N) using starch indicator. Calculate the strength of the iodine solution using the formula:

$$N_1V_1 = N_2V_2$$

where

N_1 = normality of sodium thiosulphate,

V_1 = volume of the thiosulphate consumed for 25 ml of iodine solution, in ml;

N_2 = normality of iodine solution; and

V_2 = volume of iodine solution.

6.4 Determination of Hydrogen Sulphide

Transfer the contents of the first three absorbers into a 500 ml Erlenmeyer flask. Rinse the absorbers with little distilled water. Collect the rinsed water into the same flask and then add 25 ml standardized iodine solution and 5 ml of concentrated hydrochloric acid. Wait for 10 min and then titrate the excess iodine with standard sodium thiosulphate solution using starch indicator. Run a blank simultaneously with 90 ml cadmium chloride solution to which 25 ml of standardized iodine solution and 5 ml concentrated hydrochloric acid are added. Wait for 10 min and titrate the excess iodine with standard sodium thiosulphate solution using starch indicator.

6.5 Determination of Carbon Disulphide

Transfer the contents of last two absorbers into a 500 ml Erlenmeyer flask. Rinse the absorbers with little alcohol. Neutralize the content with 5 percent acetic acid using the phenolphthalein indicator. The acid solution must be added slowly keeping the flask immersed in an ice bath during neutralization. After neutralization add starch and immediately titrate with standardized iodine solution. For clear end point, dilute the content with 4 times volume of ice cooled distilled water. Run a blank taking 60 ml alcoholic potassium hydroxide in place of absorber content and following exactly the above procedure.

NOTE — Alcoholic potassium hydroxide solution should be fresh every time. During neutralization of potassium ethylxanthate with acetic acid and prior to titration with iodine the solution should be kept in ice bath.

7 CALCULATIONS

7.1 Hydrogen Sulphide

Subtract the volume of sodium thiosulphate solution consumed from that required for a blank. Find out the equivalent volume of iodine solution to this difference. This volume is the amount of iodine solution consumed.

Concentration of

$$H_2S, \text{ ppm, } v/v = (17/1\,000) \times A_1 \times B_1 \times (24\,450/34) \times (1/1\,000) \times (10^6/V)$$
$$= (12\,400 \times A_1 \times B_1)/(V \times F)$$

where

A_1 = volume of iodine solution consumed;

B_1 = normality of the iodine solution:

V = volume of the air sample passed, in litres; and

F = dryness factor calculated from barometric pressure and temperature.

$$= (P_B - f)/P_B$$

where

P_B = barometric pressure, and

f = aqueous tension.

Aqueous tension values at different temperatures are given in Table 1.

Table 1 Water Vapour Pressure (Aqueous Tension) at Various Temperatures

Temperature °C	Vapour Pressure		Temperature °C	Vapour Pressure	
	(mm of Hg)	(millibar)		(mm of Hg)	(millibar)
(1)	(2)	(3)	(1)	(2)	(3)
16.0	13.6	18.1	28.0	28.3	37.7
18.0	15.5	20.6	30.0	31.8	42.4
20.0	17.5	23.3	32.0	35.7	47.6
22.0	19.8	26.4	34.0	39.9	53.2
24.0	22.4	29.4	36.0	44.6	59.5
26.0	25.2	33.6	38.0	49.7	66.3

7.2 Carbon Disulphide

Subtract the volume of sodium thiosulphate solution consumed from that required for a blank. Find out the equivalent volume of iodine solution to this difference. This volume is the amount of iodine solution consumed.

Concentration of

$$CS_2 \text{ (ppm, } v/v) = (76/1\,000) \times A_2 \times B_2 \times (24\,450/76) \times (1/1\,000) \times (10^6/V)$$
$$= (24\,800 \times A_2 \times B_2)/(V \times F)$$

where

A_2 = volume of iodine solution consumed, in ml;
B_2 = normality of the iodine solution;
V = volume of the air sample passed, in litres; and
F = dryness factor calculated from barometric pressure and temperature (*see* 7.1).

8 PRECISION

The precision of this standard is not established due to the difficulties in handling samples and the reactivity of hydrogen sulphide with the sample vessels.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Environment Protection and Waste Management Sectional Committee, CHD 32

Organization	*Representative(s)*
In personal capacity (*40/14, C. R. Park, New Delhi 110019*)	Prof Dilip Biswas (***Chairman***)
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Member Secretary
SHRI N. K. PAL
Scientist 'E' (Chemical), BIS

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